



# WORK PLAN FOR ADDITIONAL GEOCHEMICAL TESTING OF LISBON VALLEY MINE WASTE **ROCK**

Prepared for:



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#### 1. INTRODUCTION

The following work plan describes the protocol for additional geochemical testing of Lisbon Valley Mine waste rock. Substantial geochemical testing has been performed previously (Table 1) to assess the potential impact to groundwater from proposed backfilling in the Centennial Pit. Additional geochemical characterization has been requested by the United States Department of Interior Bureau of Land Management, Moab Field Office (BLM) and their technical consultants.

Additional testing will include column leaching tests on waste rock, specifically Bed 14 and Bed 15 materials proposed for backfill in the Centennial Pit. The column leaching tests are intended to meet the concerns expressed by BLM and their technical consultants regarding the ability of meteoric water mobility procedure (MWMP) tests to characterize potential geochemical releases from saturated backfill, and to specifically address issues of contact time and water:rock ratio. In addition to the column leaching tests requested by BLM, Lisbon Valley Mining Company's (LVMC's) technical consultants have recommended associated characterization tests on the same samples, including acid-base accounting (ABA), MWMP tests, whole rock elemental analysis, x-ray diffraction (XRD), and scanning electron microscopy and energy dispersive x-ray spectroscopy (SEM-EDS).

Table 1. Existing Water Quality and Bed 14 and Bed 15 Geochemical Characterization for the Centennial Pit Area

Test Type	EIS (1997)	Adrian Brown Consultants (1998)	Operational Characterization (2005 - 2013) <sup>(1)</sup>	Compliance Monitoring (1998 - 2013)	ARCADIS (2012)	Total
ABA	98		18	Sept 1.	98	116 <sup>(2)</sup>
SPLP	3		<u>.</u>	_		3
MWMP Single Extraction	-	10	24	-	19	53
MWMP Multiple Extraction	-	-	-	-	2	2
Intermittent Pooled Surface Water	-	•	3	-	-	3
Groundwater Samples: Burro Canyon Aquifer MW96-7A	8		-	45	38 <sup>(3)</sup> (1996 – 2010)	53
Groundwater Samples: Burro Canyon Aquifer SLV3/PW-3	13	-	-	46	29 <sup>(3)</sup> (1995 – 2010)	59

Notes

## 1.1 Data Quality Objectives

Data quality objectives (DQOs) for the column leaching tests are to:

- 1. Provide quantitative data to predict seepage characteristics of Bed 14 and 15 waste mine rock stored in saturated environments.
- 2. Produce analytical data of known and consistent quality with documented quality assurance/quality control (QA/QC) procedures.

<sup>(1)</sup> No MWMP samples were collected for operational characterization of waste rock from Jan 2008 - Mar 2009 during the mine shutdown because no waste rock was generated.

<sup>(2)</sup> Seven of the 98 samples subjected to ABA for the EIS were composited with waste rock from other beds.

<sup>(3)</sup> Groundwater used in ARCADIS 2010 modeling evaluation includes the samples listed in the EIS and subsequent compliance monitoring.

Produce analytical data that are adequate to support National Environmental Policy Act (NEPA)
impact analyses of the proposed open pit backfilling at the Lisbon Valley Mine in San Juan
County, Utah.

## 2. TESTING REGIME

The testing regime will involve collecting samples of Bed 14 and Bed 15 rock materials from the walls of the existing Centennial Pit, Sentinel Pit, and GTO Pit and from core samples of Bed 15 in the GTO area (where Bed 15 is not currently exposed). Samples will be prepared and composited (Section 3.3 and 3.5) and three monolithologic saturated columns will be processed (Table 2). No unsaturated columns will be processed, as the MWMP tests (Table 1) are considered sufficient for characterization of meteoric water infiltrating through unsaturated waste rock.

Rock Type	Geologic Unit	Source	Weathering	Saturation
RT7	Bed 14	Centennial Pit wall	weathered	Saturated
RT7	Bed 15	Centennial Pit wall	weathered	Saturated
RT7	Bed 14 replicate	Centennial Pit wall	weathered	Saturated
RT7	Bed 14	Sentinel Pit wall	weathered	Saturated
RT7	Bed 15	Sentinel Pit wall	weathered	Saturated
RT7	Bed 14	GTO Pit wall	weathered	Saturated
RT7	Bed 15	GTO Pit wall	unweathered	Saturated

Table 2. Proposed Column Leaching Tests

The column leaching tests are designed to simulate waste rock in contact with groundwater as the Burro Canyon Aquifer rebounds from its current dewatered condition. The total sample mass required for the column tests and additional geochemical characterization is shown in Table 3 and the estimated volume is shown in Table 4.

Table 3. S	Sample Mass	Required for Proposed	Testing Regimen
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Test	Sample Required	Prepped by:	Analyzed by:
Particle Size Distribution (Sieve analysis)	1.3 kg	Whetstone	Whetstone
Acid Base Accounting (ABA)	0.1 kg	Whetstone	ACZ
Meteoric Water Mobility Procedure (MWMP)	5 kg	Whetstone	ACZ
Whole rock / elemental analysis	0.1 kg	Whetstone	ACZ
Column Leaching	20 kg	Whetstone	ACZ
X-ray Diffraction (XRD)	50-100 g	GSS	GSS
SEM-EDS	1 chunk $5-10$ cm	GSS	GSS

Table 4. Approximate Required Sample Volume

Total Volume		
Approximate total mass needed	30 kg	
Assumed density	1.9	g/cm <sup>3</sup>
Volume required	15,789	cm <sup>3</sup>
Volume required	4.17	gallons

Samples will be collected by LVMC using the procedures described in Section 3.1. The bagged rock samples will be sent to Whetstone Associates' (Whetstone's) geochemistry lab for preparation, including weighing, air-drying, and compositing as described in Sections 3.3 and 3.5. A sieve analysis will be conducted as described in Section 3.4. Representative split samples will be sent to ACZ Laboratories, Inc. (ACZ), a Utah-certified and NELAC-certified laboratory, for ABA and MWMP analysis as described in Section 3.6. Samples will be submitted to GeoSystems Services (GSS) for XRD and SEM-EDS as described in Section 3.7. The saturated columns will be set up and run by Whetstone using the column testing protocols described in Section 4 and the leachates will be submitted to ACZ for analysis.

#### 3. SAMPLE COLLECTION AND PREPARATION

## 3.1 Rock Sample Collection

Rock samples from Bed 14 and Bed 15 in the exposed Centennial pit wall will be collected by LVMC using standard "channel sampling" protocols. The samples will be selected to represent "weathered" conditions and will be spatially distributed along the "outcrop" in the pit wall. The locations will be mapped, photographed, and documented with field notes, and a map will be prepared showing the sample locations. Both Bed 14 and Bed 15 have been fully characterized previously, and are known to exhibit very little spatial variability across the area of interest.

Each sample will be labeled separately and sent to the geochemistry lab for weighing, drying, and compositing as described in Section 3.3 and 3.5.

#### 3.2 Groundwater Collection

Groundwater from well PW-3 or PW-4 will be used as the head solution for column testing. The major ion water quality is similar for both wells (Figure 1). PW-3 is a 6-inch well completed to a depth of 476 feet in the Burro Canyon Aquifer, adjacent to the Centennial Pit (Figure 2). PW-4 is a 6-inch well completed to a depth of 430 feet the Burro Canyon Aquifer between the Sentinel and Centennial Pits.

Redox conditions have not been directly determined for the Burro Canyon Aquifer to date; however, reducing conditions are anticipated based on the high average dissolved iron suggesting ferrous iron (Fe<sup>2+</sup>) and low dissolved aluminum concentrations suggesting suggest the iron concentrations are in fact due to dissolved ferrous iron rather than nanoscale ferric iron colloids. Therefore, precautions will be taken to collect a representative solution and prevent contact with atmospheric oxygen.

The Burro Canyon groundwater will be collected as follows:

- Connect to the existing 3-inch camlock fitting (do not use the ¼ inch sampling port)
- Assemble clean stainless steel, alumimum, or PVC fittings that telescope from the 3-inch camlock fitting to a 3/4-inch or 1-inch flexible hose.
- Insert the flexible hose into the bottom of a clean 5-gallon carboy, a 1-gallon cubitainer, or similar container.
- Insert a dissolved oxygen probe into the carboy or cubitainer.
- Turn on the dedicated submersible pump. Allow the well to pump into the raw water system for at least 5 minutes prior to opening the valve to the 3-inch port. The current pumping rate at PW-3 is approximately 10-15 gpm, while PW-4 is pumped at 30 gpm.
- Fill the carboy from the bottom to the top and allow the water to overflow the top of the carboy until the dissolved oxygen is stable.
- Cap the carboy immediately with zero headspace.

- Collect a total of eight (8) gallons per column using this procedure (24 gallons for three columns).
- Collect an additional sample for ACZ analysis of head solution at the same time and submit the sample directly to ACZ with the standard GW analytical suite. This sample may also be used as the 4<sup>th</sup> Quarter compliance monitoring sample.

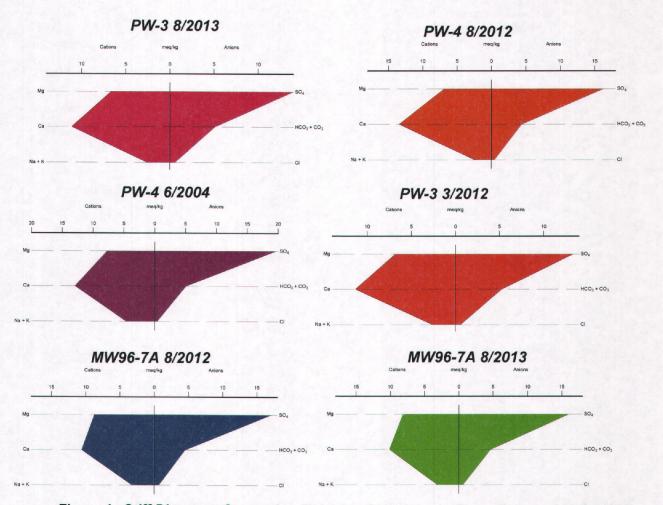


Figure 1. Stiff Diagrams Comparing Major Ion Chemistry in Burro Canyon Aquifer Wells

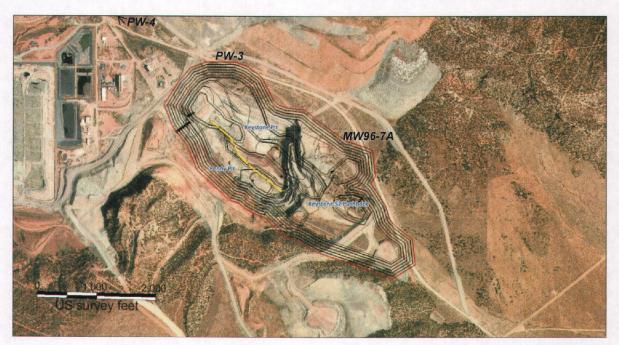


Figure 2. Location of Burro Canyon Aquifer Wells PW-3, PW-4, and MW96-7A

## 3.3 Sample Preparation

Waste rock samples for column leaching tests will be air-dried as-received at room temperature ( $21^{\circ}C \pm 3^{\circ}$ ) to stable weight and logged for lithology, mineralogy, and weathering alteration. After drying, the entirety of each sample will be screened to pass a 0.75-inch wire mesh. Oversized material will be reduced by hand breaking or jaw crushing to achieve a column diameter to maximum particle size ratio of 8:1. A representative split of the screened sample will be prepared using methodology described in ASTM 702-98 and set aside for standard particle size analysis using mesh sieves no. 5, 10, 18, 35, 60, 120, and 230.

Sample preparation by Whetstone will consist of the following:

- Log in and weigh the sample
- Record the sample ID and wet/received weight in the lab book designated for the project.
- · Fill a chip tray and characterize each sample for lithology, mineralogy, and weathering
- Lay the sample on plastic and air dry at room temperature  $(21^{\circ}C \pm 3^{\circ})$  to stable weight (typically 2 3 days)
- Weigh each sample and record the dry weight
- Screen the entirety of each sample to pass a 0.75-inch sieve.
- Reduce any oversized material to -3/4 inch using a jaw crusher or by hand (if material is suitably friable/breakable)
- Prepare composite sample based on equal mass of all individual samples of the specific rock unit
- Cone and quarter or use finger splitter to extract representative samples (Section 3.5)
- Submit 50 g and 5 kg splits to ACZ Laboratories for ABA and MWMP analysis
- Split 20 kg for column leaching test in accordance with protocols (Section 4)
- Perform sieve analysis

## 3.4 Particle Size Distribution (Sieve Analysis)

A representative split of approximately 1300 grams (g) of the composited sample will be prepared using methodology described in ASTM 702-98 and set aside for standard particle size analysis using mesh sieves no. 5, 10, 18, 35, 60, 120, and 230. The procedures for sieve analysis are as follows:

- 1. Weigh the entire dried sub sample and record the sample mass.
- 2. Organize the screens in the appropriate order (bottom pan, 230, 120, 60, 35, 18, 10, 5, 3/4 in, lid).
- 3. Use a mortar and rubber-tipped pestle to break up particles and separate coarse and fine particles. Do not break up rock fragments.
- 4. Pour sample from its container onto top sieve, taking care to minimize the creation of dust.
- 5. Cover the stack of sieves with the lid and place in sieve shaker.
- 6. Shake for 20 minutes, or until one mass percent of the residue on a sieve passes that sieve during one minute of sieving.
- 7. Remove the lid from the sieve set and verify no material was retained on the top sieve.
- 8. Mortar and pestle any sample retained on the top sieve and each sieve smaller than No. 10 to ensure proper particle size distribution. Repeat steps 4-6.
- 9. Remove the next sieve and turn the sieve upside down so the retained material falls onto the transfer container without spillage or creating dust. Remove any material remaining in the sieve using a sieve brush. Transfer this material into the cumulative mass container and record the mass contained in the cumulative mass container.
- 10. Continue for the remaining sieves and bottom pan.
- 11. Calculate percent passing for each sieve.

## 3.5 Sample Compositing and Splitting

Cone-and-quarter compositing and splitting are performed by gathering the sample into a pile (cone), digging out the center of the cone and distributing the material around the edges of the pile in a ring, and gathering the ring back into a center cone. This procedure homogenizes the sample and is repeated three times. The cone is then split into equal quarters and opposite quarters are combined to produce a representative split. Sequential generations of splits are combined until the sample for the composite is within 0.1 gram of the target weight. The resulting composited samples are believed to be representative of the materials of interest. Splits of each composite are sent to ACZ and GeoSystems Services (GSS) for analysis, with a request to process and analyze them in their entirety, and used in Whetstone's geochemistry lab. These procedures are implemented to avoid potential sample bias related to shipping, settlement, and unrepresentative splitting at the analytical lab.

#### 3.6 ABA, MWMP and Elemental Analyses

The representative splits will be sent to ACZ Laboratories and analyzed for the suite of constituents listed in Table 5 and Table 6. Numerous MWMP and ABA tests have been performed to date (Table 1). ABA is a quick and relatively low cost chemical test used to estimate the capacity of material to produce and neutralize acid by comparing the acid generating potential (AGP) with the acid neutralization potential (ANP) for a given material.

Table 5. Laboratory Analytical Suite for LVMC MWMP Leaching Tests

Parameter	Analytical Method	Method Detection Limit	Units
Major Ions and Solution Parameters			
pH	SM 4500H+B	0.1	s.u.
Alkalinity, Total as CaCO <sub>3</sub>	SM 2320B	2	mg/L
Alkalinity, Carbonate as CaCO <sub>3</sub>	SM 2320B	2	mg/L
Alkalinity, Bicarbonate as CaCO <sub>3</sub>	SM 2320B	2	mg/L
Alkalinity, Hydroxide as CaCO <sub>3</sub>	SM 2320B	2	mg/L
Hardness as CaCO <sub>3</sub>	SM 2340B	1.5	Calculation
Calcium	EPA 6010B	0.2	mg/L
Magnesium	EPA 6010B	0.2	mg/L
Potassium	EPA 6010B	0.3	mg/L
Sodium	EPA 6010B	0.3	mg/L
Chloride	EPA 300.0	0.5	mg/L
Fluoride	EPA 300.0	0.1	mg/L
Sulfate	EPA 300.0	0.5	mg/L
Phosphorus	SM365.1	0.01	mg/L
Silica	M200.7	0.428	mg/L
Residue, Filterable (TDS) @180C	SM 2540C	10	mg/L
TDS, Calculated	Calculation		mg/L
Specific Conductance @25C	SM 2510B	1	umhos/cm
Anion-Cation Balance	Calculation	_	
Metals – Dissolved			
Aluminum	EPA 6010B	0.03	mg/L
Antimony	EPA 6020A	0.0004	mg/L
Arsenic	EPA 6020A	0.0002	mg/L
Barium	EPA 6010B	0.003	mg/L
Beryllium	EPA 6020A	0.00005	mg/L
Cadmium	EPA 6020A	0.0001	mg/L
Chromium	EPA 6020A	0.0005	mg/L
Copper	EPA 6020A	0.0005	mg/L
Iron	EPA 6010B	0.02	mg/L
Lead	EPA 6020A	0.0001	mg/L
Manganese	EPA 6020A	0.0005	mg/L
Mercury	EPA 7470A	0.0003	mg/L
Molybdenum	EPA 6010B	0.002	mg/L
Nickel	EPA 6010B	0.01	mg/L
Selenium	EPA 6020A	0.0001	mg/L
Silver	EPA 6020A	0.0001	mg/L
Thallium	EPA 6020A	0.0003	mg/L
Uranium	EPA 6020A	0.0001	
Vanadium	EPA 6020A	0.0001	mg/L
Zinc		0.0002	mg/L
	EPA 6020A	0.002	mg/L
Metals – Total Uranium	EPA 6020A	0.0001	mg/L

Table 6. Laboratory Analytical Suite for ABA Tests

Parameter	Method
Acid Generation Potential (calc on Sulfide Sulfur)	M600/2-78-054 1.3
Acid Neutralization Potential (calc)	M600/2-78-054 1.3
Acid-Base Potential (calc on Sulfur total)	M600/2-78-054 1.3
Neutralization Potential as CaCO <sub>3</sub>	M600/2-78-054 3.2.3
Sulfur Forms (Total, Sulfide, Sulfate, Insoluble)	M600/2-78-054 3.2.4-MOD
Acid Generation Potential (calc on Sulfide Sulfur)	M600/2-78-054 1.3

Table 7. Laboratory Analytical Suite for Elemental Analysis

Parameter	Units
Metals - Dissolve	ed
Aluminum	mg/kg
Antimony	mg/kg
Arsenic	mg/kg
Barium	mg/kg
Beryllium	mg/kg
Cadmium	mg/kg
Chromium	mg/kg
Copper	mg/kg
Iron	mg/kg
Lead	mg/kg
Manganese	mg/kg
Mercury	mg/kg
Molybdenum	mg/kg
Nickel	mg/kg
Selenium	mg/kg
Silver	mg/kg
Thallium	mg/kg
Uranium	mg/kg
Vanadium	mg/kg
Zinc	mg/kg

#### 3.7 XRD and SEM-EDS

Samples for XRD and SEM-EDS will be sent to GSS for mineralogical analysis. GSS will perform XRD with clay mineralogy, general thin section analysis, and EDS spectral mapping.

X-ray diffraction (XRD) of bulk powdered rock material is recommended for quantitative assessment of mineralogy. Quantitative XRD yields weight percent abundance of the different mineral phases present, down to approximately 1% by weight. The XRD analysis will primarily be used to confirm major mineral phases (e.g., carbonate and sulfide minerals) and distinguish and quantify clay minerals (such as smectite, illite, chlorite, kaolinite, etc). The petrographic analysis will include preparation of a rock specimen as a thin section which will then be examined with a petrographic microscope (transmitted light) to further characterize the different mineral phases by their optical properties. GSS will also examine a polished specimen by electron microscopy in both backscattered electron and EDS x-ray spectroscopy modes. EDS analysis is x-ray fluorescence using the electron beam of the electron microscope as a low energy source.

#### 4. COLUMN TESTING PROTOCOL

Monolithologic columns are recommended for this study over run of mine (ROM) columns packed with a mixture of rock types that are proportioned to represent the material balances of the proposed backfill scenarios. The primary advantage of monolithologic columns is the ability to obtain the individual drainage chemistry information associated with Bed 14 and Bed 15 waste rock, which can be visually identified as individual rock types that may be selectively handled. ROM columns provide an assessment of drainage quality of a discrete rock mixture but do not have the advantage of monolithologic columns that allow adjustment of water quality predictions if waste rock material balances are updated due to adjustments to the mine plan.

## 4.1 Scope and Applicability

- i. This method is a standard column testing procedure for generating aqueous leachates from Bed 14 and Bed 15 of the Lisbon Valley Mine. It is applicable to saturated waste rock, including (but not limited to) saturated pit backfill.
- ii. Although microorganisms are known to mediate redox reactions involving sulfide minerals in waste rock and ore, such sulfide minerals are scarce or absent in the Bed 14 and Bed 15 material to be tested. Therefore, the following protocol specifies that the columns are not to be inoculated with bacteria or sterilized to eliminate bacteria that naturally exist in the solid sample material.
- iii. This method is intended to provide site-specific leaching data to support impact analyses and decision-making under NEPA.
- iv. This method provides leachates that are suitable for analysis of nonvolatile compounds and solution parameters including major ions, metals, and metalloids.
- v. Leachates produced by the test may not be in chemical equilibrium with the solid materials contained within the column.
- vi. Analytical data from the column effluents provide information about the leaching characteristics of materials under the conditions used in the test and are not intended to be the sole basis for characterization of the materials, determination of environmental mobility of specific constituents, or engineering design of mine facilities.

## 4.2 Summary of Test Method

This column testing method is designed to evaluate the kinetic leaching characteristics of a 20 kg sample of waste rock under saturated conditions. The leaching head solution will be groundwater from the Burro Canyon Aquifer. The testing method produces approximately 5,000 ml of effluent during each leaching cycle. The effluent is suitable for analysis of solubilized nonvolatile constituents to determine the release characteristics of the solid material under the test conditions.

The test is designed to simulate Burro Canyon Aquifer groundwater coming in contact with the pit backfill. The Burro Canyon Aquifer is currently dewatered to well below the pit floor and will rise after mining when the pumps are shut off. As the aquifer recharges, groundwater levels will rise to a level within the pit backfill (and below the surface of the proposed backfill). The columns will simulate flooding of the backfill from below. The Burro Canyon Aquifer has been designated as a Class III Aquifer by the Utah Department of Environmental Quality (UDEQ, 1998), which indicates that total dissolved solids (TDS) are greater than 3,000 mg/l and less than 10,000 mg/l, or one or more constituents exceed the ground water quality standards.

The test is performed in a 6-inch diameter cylindrical column. Multiple columns may be configured in parallel to permit simultaneous testing of several samples. The test procedure specifies repeated leaching cycles consisting of a solution application period and a reaction period. The column is operated under upward flow conditions by applying the head solution to the bottom of the column and collecting the effluent from the top.

# 4.3 Analytical Suite for Column Leaching Tests

The analyte list for column leaching studies includes major ions, COPCs, and other solution parameters needed to evaluate compliance with water quality standards contained in Utah Administrative Code (UAC) R317-6-2 and to assess long-term water quality. The analyte list for column test leachates is shown in Table 8.

Table 8. Laboratory Analytical Suite for LVMC Column Leaching Tests

Parameter	Analytical Method	Method Detection Limit	Units
Major Ions and Solution Parameters			
pH	SM 4500H+B	0.1	s.u.
Alkalinity, Total as CaCO <sub>3</sub>	SM 2320B	2	mg/L
Alkalinity, Carbonate as CaCO <sub>3</sub>	SM 2320B	2	mg/L
Alkalinity, Bicarbonate as CaCO <sub>3</sub>	SM 2320B	2	mg/L
Alkalinity, Hydroxide as CaCO <sub>3</sub>	SM 2320B	2	mg/L
Hardness as CaCO <sub>3</sub>	SM 2340B	1.5	Calculation
Calcium	EPA 6010B	0.2	mg/L
Magnesium	EPA 6010B	0.2	mg/L
Potassium	EPA 6010B	0.3	mg/L
Sodium	EPA 6010B	0.3	mg/L
Chloride	EPA 300.0	0.5	mg/L
Fluoride	EPA 300.0	0.1	mg/L
Sulfate	EPA 300.0	0.5	
Phosphorus	SM365.1	0.01	mg/L
Silica			mg/L
	M200.7	0.428	mg/L
Residue, Filterable (TDS) @180C	SM 2540C	10	mg/L
TDS, Calculated	Calculation	-	
Specific Conductance @25C	SM 2510B	1	umhos/cm
Anion-Cation Balance	Calculation	<u> </u>	
Metals – Dissolved			
Aluminum	EPA 6010B	0.03	mg/L
Antimony	EPA 6020A	0.0004	mg/L
Arsenic	EPA 6020A	0.0002	mg/L
Barium	EPA 6010B	0.003	mg/L
Beryllium	EPA 6020A	0.00005	mg/L
Cadmium	EPA 6020A	0.0001	mg/L
Chromium	EPA 6020A	0.0005	mg/L
Copper	EPA 6020A	0.0005	mg/L
Iron	EPA 6010B	0.02	mg/L
Lead	EPA 6020A	0.0001	mg/L
Manganese	EPA 6020A	0.0005	mg/L
Mercury	EPA 7470A	0.0002	mg/L
Molybdenum	EPA 6010B	0.01	mg/L
Nickel	EPA 6010B	0.01	mg/L
Selenium	EPA 6020A	0.0001	mg/L
Silver	EPA 6020A	0.00005	mg/L
Thallium	EPA 6020A	0.0001	mg/L
Uranium	EPA 6020A	0.0001	mg/L
Vanadium	EPA 6020A	0.0001	mg/L
Zinc	EPA 6020A	0.002	mg/L mg/L
Metals – Total			
Uranium	EPA 6020A	0.0001	mg/L
	D171 002071	0.0001	mg/L
"Field" Parameters Temperature		0.1	
pH		0.1	
		0.1	
Specific Conductance (E.C.) ORP			
Dissolved oxygen (D.O.)			

Notes: Whetstone lab monitors "field parameters" on every PV cycle (or half cycle, as required)

## 4.4 Apparatus

#### 4.4.1 Column

The column body is be constructed of clear polyvinyl chloride (PVC) or polycarbonate pipe, approximately 36 inches in length, with an inside diameter of 6 inches and a minimum wall thickness of 0.280 inches (Figure 3). An opaque PVC end cap with centered solution application port and 1/4-inch diameter 316 stainless steel ball valve is fixed to the bottom of the column body using PVC cement or other sealant to ensure that the connection is watertight. Because PVC cement and other sealants have the potential to affect leachate chemistry, an equipment blank sample will be prepared and evaluated for the column prior to loading. An opaque PVC end cap with centered sample collection port and 1/4-inch diameter 316 stainless steel ball valve with barbed tubing fitting is installed at the top of the column body after the column has been loaded with the solid sample material. The top cap will be sealed around the exterior bottom edge with silicone caulk to prevent leakage. PVC cement or other sealants applied to the interior of the cap will be avoided to minimize the risk of contamination of column leachates by volatile organic carbon (VOCs) vapors.

## 4.4.2 Inert Column Packing Material

Ottowa inert clean silica sand packing material is placed at the top and bottom of each column to elevate the sample above the opaque PVC cap. The silica sand is decontaminated with a de-ionized water wash and allowed to dry completely before use in the column. Based on previous column experiments conducted using the Ottowa silica sand, the leachate concentrations associated with the silica sand are low compared to concentrations in the head solution. The packing material will be well-graded.

## 4.4.3 Metering Pump and Reagent Water Supply

A metering pump capable of accurately delivering 15 to 300 ml/hr (± 3%) is required to apply head solution water to the bottom of the column. When multiple columns are operated simultaneously, each column should have a separate metering pump connected to a common head solution supply. Tubing from the head solution supply may be configured in series or by manifold to supply multiple pumps. Tubing and vessels that are used to convey or store head solution will be constructed of inert material such as glass, polyethylene, Teflon<sup>®</sup>, or Tygon<sup>®</sup>. Valves and other tubing fittings will be 316 stainless steel, polyethylene, or other inert material. All tapered threads on valve bodies and tubing connections will be sealed with Teflon<sup>®</sup> tape to prevent leakage.

#### 4.5 Procedure

## 4.5.1 Column Decontamination and Preparation of Equipment Blank Sample

The column apparatus will be thoroughly decontaminated prior to sample placement using the following procedure. The column and associated fittings are scrubbed with a non-ionic surfactant detergent (Liquinox® or Alconox®) and tap water to remove gross contamination from surfaces, followed by an acid wash with a 10% solution of reagent-grade nitric acid and de-ionized water, and a final triple rinse with de-ionized water. The apparatus will be permitted to air dry. After decontamination, an equipment blank sample will be prepared for each column. An equipment blank sample consists of 5,000 ml of reagent water poured down the interior wall of the column to contact as much of the surface as possible. The sample will be allowed to stand in the column for 24 hours before collection and analysis of the parameters listed in Table 8.

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<sup>&</sup>lt;sup>1</sup> In many cases, contamination issues for total organic carbon (TOC) may be avoided if PVC cement and sealants are allowed sufficient time to cure and de-gas before the column is put into service.

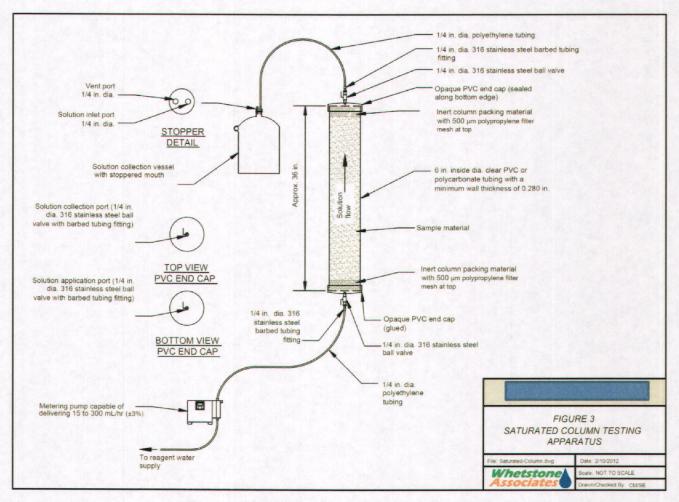


Figure 3. Saturated Column Testing Apparatus

#### 4.5.2 Solid Sample Preparation

As described previously in this Work Plan, rock samples for column leaching tests will be air-dried asreceived at room temperature ( $21^{\circ}C \pm 3^{\circ}$ ) to stable weight and logged for lithology, mineralogy, and weathering alteration. After drying, the entirety of each sample will be screened to pass a 0.75-inch wire mesh with oversized material being reduced by hand breaking or jaw crushing to achieve a column diameter to maximum particle size ratio of 8:1. A representative split of the screened sample will be prepared using methodology described in ASTM 702-98 and set aside for standard particle size analysis using mesh sieves no. 5, 10, 18, 35, 60, 120, and 230.

#### 4.5.3 Column Packing

Sample and construction materials will be placed in the column in the following order:

- 1. A 6-inch diameter disk of polypropylene filter mesh with a pore opening of 500 μm will be placed in the bottom of the column.
- 2. A 4-inch thick layer of inert column packing material will be placed over the polypropylene filter mesh to elevate the sample above the opaque end cap.

- 3. The solid sample material (20 kg) will be placed above the inert column packing material in random lifts of varying thickness. Material in the columns will be compacted between lifts by gently tapping on the side of the column with a rubber mallet. The surface of each lift will be scarified (roughened) prior to the placement of the next lift to minimize the potential for preferential flow along the contact between lifts. Individual lifts should not have a thickness exceeding 3 inches.
- 4. The remaining volume of the column (typically 4- to 6-inches) will be filled with inert column packing material.
- 5. A 6-inch diameter disk of polypropylene filter mesh with a pore opening of 500 µm will be placed over the top of the inert packing material to limit solid sample loss in the effluent.
- 6. The porosity of each column will be determined by measuring height of rock material in clear PVC column (not including the inert packing material).
  - a. Calculate column volume:  $Vc = \pi D2/4h$
  - b. Calculate material volume based on density  $Vr = 20 \text{ kg} * \rho$ 
    - i. Where  $\rho = \text{density in gm/cm}3$
  - c. Calculate porosity n = Vc/Vr

## 4.5.4 Column Operation

#### 4.5.4.1 General

A common water supply reservoir will be used for multiple columns that are configured to permit simultaneous testing. The reagent solution (groundwater from Burro Canyon Aquifer well PW-3 or PW-4) will be kept free of contact with atmospheric oxygen. Laboratory analysis of the head solution used in the tests will be submitted for analysis of the parameters in Table 8. The columns will be maintained at room temperature  $(21^{\circ}C \pm 3^{\circ})$  during the testing period, and the laboratory kept dark except during leachate collection and column maintenance to minimize the potential for photo-oxidation of metallic constituents.

The head solution will be applied at a rate of 15 ml/hr ( $\pm$  3%) = 0.25ml/min. This is the lowest practical application for the metering pumps, which is required to prevent column plugging. After initial column wetting, a three-day static contact period will be allowed before pore volume samples are run and collected. Collect samples from the first 0.5 pore volume (PV-0.5), first pore volume (PV-1) and second pore volume (PV-2). One pore volume moves through the column in approximately 10 days, depending on column porosity (to be determined in Table 9) so PV-0.5 will be collected on Day 5 of head solution application. An automatic solenoid switch will be used to obtain PV-0.5.

Table 9. Approximate Column Leaching Rate

Column Inner Diameter	6	inches
Column Length	30	inches
Total Volume	848.2	in <sup>3</sup>
Total Volume	13.9	L
Effective porosity	0.25	
Effective pore volume	3.475	L
Flow rate	15	mL/hr
	0.36	L/day
Column Residence Time	9.65	days
Pore Volumes per Day	0.1036	days-1

Notes: The above table provides scoping calculations only.

Sample mass is fixed at 20 kg. Column length is determined by sample density and porosity (Table 10).

Table 10. Forward Calculation of Column Length

Sample size	20	kg
Rock density		gm/cm <sup>3</sup>
Rock matrix volume	10,526.3	cm <sup>3</sup>
Rock matrix volume	642.4	in <sup>3</sup>
Porosity	30%	
Total column volume	835.1	in <sup>3</sup>
Column height	29.53	inches

Additional groundwater in excess of the 5,000 ml target effluent volume is applied to the bottom of the column during the first leaching cycle to compensate for the volume of water that is retained by the column during saturation of the sample and inert packing material. The required additional volume is variable between columns and depends on the characteristics of the sample. Experimental data indicates that columns may retain between 7,000 and 8,000 ml of the solution applied during the first cycle (Whetstone, 2010).

Column leachates will be collected in a clean container that has been washed with a 10% solution of reagent-grade nitric acid and de-ionized water, triple rinsed with de-ionized water, and allowed to dry. The volume, temperature, pH, DO, ORP, and EC of the effluent solution will be measured at the time of collection and the leachates will be filtered  $(0.45 \, \mu m)$  and preserved as appropriate for the analytical suite and prior to shipment to the analytical laboratory. The column will be visually monitored daily for evidence of channelized flow, bacterial activity, and iron or manganese oxide formation. A written record of column maintenance, performance, and observations, will be kept in the laboratory record.

### 4.5.4.2 First Cycle Operation

The first leaching cycle is designed to produce  $5,000 \text{ ml} (\pm 5\%)$ , or approximately one pore volume, of effluent and will require approximately 16 days to complete. It includes an initial solution application period of approximately 1 day to saturate the column, a 3-day reaction period, and a 10-day solution application period to generate effluent for chemical analysis. The column effluent is collected in two aliquots representing the first (PV-0.5) and second (PV-1) half pore volumes of solution released from the column.

The initial solution application period to saturate the column requires approximately 1 day to complete. Groundwater will be added to the column through the solution application port at a rate of 200 ml/hr ( $\pm 1\%$ ). The column will be observed frequently to track the saturation level as it rises, and the application period will be stopped as close as practical to the point when the first drop of effluent is released from the column into the solution collection vessel. The total volume of solution applied will be recorded in the experimental record along with the date and time of the first effluent and other observations made during column saturation.

Following saturation, the column will be allowed to stand idle for 3 days (72 hrs) to permit the solution to react with the solid sample. The duration of the reaction period may be adjusted  $\pm$  6 hours to facilitate scheduling of multiple columns for the next phase of operation.

The duration of the solution application period to generate effluent for chemical analysis is expected to be 10 days (240 hours), during which 3,600 ml of reagent water will be added to the column through the solution application port at a rate of 15 ml/hr ( $\pm 3\%$ ). The column will be observed daily for evidence of preferential flow, bacterial activity (biofilms) or mineral precipitates. Observations relevant to column operation and adjustments to the solution application rate will be recorded in the experimental record. The effluent from the first cycle will be collected in two aliquots. Effluent volume, temperature, pH, DO, ORP, and EC will be measured for each aliquot and the samples will be filtered (0.45  $\mu$ m) and preserved as

appropriate prior to shipping to the analytical laboratory for the analytes listed in Table 3. The first aliquot of 1,800 ml will be collected at 5 days (120 hrs) from the start of the solution application period. The second aliquot of 1,800 ml will be collected at end of the solution application period. The solution collection vessel will be emptied, decontaminated, and replaced between each sample that is collected for analysis.

## 4.5.4.3 Subsequent Cycle Operation

Each leaching cycle after the first cycle will require 10 days to complete and will include a reaction period (3 days) and a solution application period (10 days).

The column will be allowed to stand idle for 3 days (72 hrs) after the first leaching cycle to permit the solution to react with the solid sample.

The groundwater solution application period starts at the end of the reaction period and is expected to have a duration of 10 days (240 hours). The application rate will be 15 ml/hr ( $\pm 1\%$ ) to add a total of 3,600 ml of reagent water to the column during each leaching cycle. The column will be observed daily for evidence of preferential flow, bacterial activity (biofilms) or mineral precipitates. Observations relevant to column operation and adjustments to the solution application rate will be recorded in the experimental record. At the end of the application period, effluent volume, temperature, pH, DO, ORP, and EC will be measured and the samples will be filtered (0.45  $\mu$ m) and preserved as appropriate prior to shipping to the analytical laboratory for the analytes listed in Table 8. The solution collection vessel will be emptied, decontaminated, and replaced in preparation for the next leaching cycle.

## 4.5.5 Duration of Testing

Column tests will be performed for two leaching cycles (approximately 26 days).

## 4.6 Quality Control for Column Leaching Tests

In addition to standard laboratory QA/QC procedures associated with the analytical methods listed in Table 8, the column leaching study will incorporate replicate columns, equipment blanks, and blind duplicate split samples as described below.

#### 4.6.1 Replicate Columns

Replicate columns provide QC for the evaluation of experimental precision and reproducibility. A replicate column is an exact duplicate of another column and is operated under identical conditions. One replicate column will be prepared and leachates from replicate column will be analyzed for the same suite of constituents as the original column.

#### 4.6.2 Equipment Blank Samples

Equipment blank samples are used to assess potential contamination from the testing apparatus and from the sample preparation and analytical procedures. Equipment blank samples will be prepared for each column prior to the start of testing, a total of three blanks are required for this study. The column apparatus will be scrubbed with a non-ionic surfactant detergent solution to remove gross contamination, followed by an acid wash with a 10% solution of reagent-grade nitric acid and de-ionized water and triple rinsing with de-ionized water. After decontamination, the equipment blank sample will be prepared by adding 5,000 ml of reagent water to the column and allowing it to react for 24 hours before collection. The equipment blank sample will be analyzed for the same suite of constituents as the column leachates.

An equipment blank will also be prepared for packing material (Ottowa silica sand) that is used to elevate the sample above the bottom end cap in the columns or distribute the head solution at the top of the columns. The packing material will be decontaminated by washing in tap water to remove gross

contamination followed by triple rinsing with de-ionized water. The packing materials will be allowed to dry completely before preparing the equipment blank. The equipment blank is prepared by placing 10 kg of packing material in contact with 2,500 ml of reagent water and allowing it to react for 24 hours before collection.

## 4.6.3 Blind Duplicate Samples

Blind duplicate samples are used to assess analytical precision and consistency of the sample preparation process. One blind duplicate sample will prepared for a randomly selected column during the study and submitted for laboratory analysis. A blind duplicate sample is prepared by splitting a leachate into two or more aliquots prior to sample preparation. The samples are then carried through the preparation and analytical process. Blind duplicate samples will be submitted to the laboratory under an alias sample name and analyzed for the same suite of constituents as the original sample.

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